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JC535 U.S. PTO 08/16/00

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UTILITY	Atty Doc. No. 50634 Total Page 12
PATENT APPLICATION	FIRST NAMED INVENTOR OR APPLICATION IDENTIFIER
TRANSMITTAL	Joachim PAUST
	Express Mail Label No.

JC531 U.S. PTO 09/639681 08/16/00

Application Elements

Address To: Assistant Commissioner for Patents
Box Patent Application
Washington, D.C. 20231

1. / X / Fee transmittal Form
(Submit an original, and a duplicate for fee processing)
2./ X /Specification Total Pages /
(Preferred arrangement set for below)

Descriptive title of the Invention
Cross References to Related Application
Statement Regarding Fed. Sponsored R & D
Reference to Microfiche Appendix
Background of the Invention
Brief Summary of the Invention
Brief Description of the Drawings (if filed)
Detailed Description
Claim(s)
Abstract of the Disclosure

3./ / Drawing(s)(35 USC 113)(Figs.) Total Sheets / /
4./ X /Oath or Declaration Total Pages/ 3 /
a / X / Newly executed (original or copy)
b./ /Copy from a prior application (37 CFR 1.63(d)
(For Continuation/Divisional with Box 17 completed)
Note Box 5 below
i./ / DELETION OF INVENTOR(S)
Signed statement attached deleting inventor(s) named in the prior application see 37 CFR 1.63(d)(2) and 1.33(b).
5. / / Incorporation by reference (useable if Box 4b is checked)
The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.

6. / / Microfiche Computer Program (Appendix)
/7./ /Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
a./ / Computer Readable Copy
b/ / Paper Copy (Identical to computer copy)
c/ / Statement verifying identity of above copies

ACCOMPANYING APPLICATIONS PARTS

8./ X / Assignment Papers (cover sheet & document(s)
9/ / 37 CFR 3.73(b)Statement / /Power of Attorney
10./ /English Translation Document (if applicable)
11./ /Information Disclosure / / Copies of IDS Citations
12./ X /Preliminary Amendment
13./ x/Return Receipt Postcard (MPEP 503)
Should be specifically itemized)
14./ /Small Entity / /Statement filed in prior application
Statements Status still proper and desired
15./X/ Certified Copy of Priority Document(s)
(if foreign priority is claimed)
16./ / Other

17. If a Continuing Application, check appropriate box and supply the requisite information:
/ /Continuation / /Divisional / / Continuation-in part (CIP) of prior application No.

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The filing fee has been calculated as shown below:

For:	Number Filed	Number Extra	SMALL/LARGE ENTITY	BASIC FEE $345./$690.
Basic Fee.....				$_____
Total Claims:	15	-20 =_____	x $09./$18.	= _____
Indep. Claims:	2	-3 =_____	x $39./$78.	= _____
[ ] Multiple Dependent Claim(s) presented:			$130./260	=_____
[x] A check is enclosed for the filing fee.				$ 690.00

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[X] A check for $730.00 for the filing fee and recordation of assignment fee.

[X] The Commissioner is hereby authorized to charge any other fee required, including the issue fee, in connection with the filing and prosecution of this application, and to the extent necessary, applicant(s) hereby petition for extension(s)of time under 37 CFR 1.136, to be charged to our Deposit Account 11-0345.

Respectfully submitted,
KEIL & WEINKAUF

HBKeil

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	)	
	)	
PAUST et al.	)	Group Art:
	)	
Serial No. Not yet assigned	)	Examiner
	)	
Filed: With Application	)	
	)	

For: PREPARATION OF N-ACYLAMINO ACID ESTERS AND ACYLAMINO ACETALS

PRELIMINARY AMENDMENT

Hon. Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Prior to examination, kindly amend the above-identified application as follows:

IN THE CLAIMS

Claim 4, line 1, delete "any of claims 2 or 3" and insert --claim 2--.

Claim 7, line 1, delete "either of claims 5 or 6" and insert --claim 5--.

Claim 8, line 1, delete "any of claims 2 to 4" and insert --claim 2--.

Claim 10, line 1, delete "or 9".

Claim 11, line 1, delete "any of claims 5 to 7" and insert --claim 5--.

Claim 13, line 1, delete "either of claims 11 or 12" and insert --claim 11--.

REMARKS

The claims have been amended to eliminate multiple dependency. No new matter has been added.

Entry of the above amendment is respectfully solicited.

Respectfully submitted,

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Preparation of N-acylamino acid esters and N-acylamino acetals

The invention relates to a process for preparing N-acylamino acid esters and N-acylamino acetals.

A large number of different methods for synthesizing amino acids and their esters are known. A review is given, inter alia, in Ullmanns Encyclopedia of Industrial Chemistry, Vol. A2, 57-97, VCH Weinheim 1985.

Industrial syntheses of D,L-α-amino acids, for example the Strecker synthesis, use aldehydes as starting materials, which are reacted with NH_3 and HCN to give aminonitriles. The nitrile group can subsequently be reacted with alcohols or water to give the corresponding esters and amino acids, respectively.

DE-A-3145736 describes a process for preparing N-formyl-α-amino acid esters by reacting aminonitriles - for example from the Strecker synthesis - with an appropriate alcohol and formamide in the presence of hydrogen chloride.

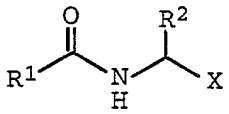
Also known is the preparation of N-formyl-D,L-alanine from pyruvic acid by boiling with ammonium formate in formic acid [F. Yoneda and K. Kuroda, J. Chem. Soc. Chem. Commun., 1982, 927-929].

N-Formylalanine esters are used, inter alia, for preparing vitamin B_6 (Pyridoxine) [Review by König and Böll, Chem. Ztg. 100 (1976), 107/8] and isocyanic acid, for example according to Ugi, Angew. Chem. 77 (1965), 492.

The processes described have the disadvantage that the starting materials used are finished amino acids or precursors thereof - for example cyanohydrins or aminonitriles from the Strecker synthesis - which have to be prepared beforehand in a separate process.

It is an object of the present invention to provide a process for preparing N-acylamino acid esters and N-acylamino acetals which can easily be carried out on an industrial scale, using readily-obtainable starting materials.

We have found that this object is achieved by a process for preparing N-acyl derivatives of the formula I



I

in which the substituents independently of one another have the following meanings:

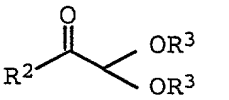
X is $CH(OR^3)_2$, $COOR^3$;

R^1 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^2 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^3 is C_1-C_{12}-alkyl,

which comprises reacting a carboxamide R^1-$CONH_2$ of the formula II with a glyoxal monoacetal derivative of the formula III,



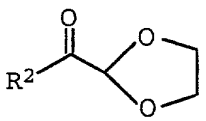
III

in the presence of a carboxylic acid R^4-COOH of the formula IV where R^4 = C_1-C_{12}-alkyl, where the substituents R^1 to R^3 are as defined above.

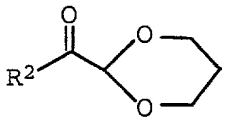
Alkyl radicals for R^1 to R^4 which may be mentioned are branched or straight-chain C_1-C_{12}-alkyl chains, for example methyl, ethyl, n-propyl, 1-methylethyl, n-butyl, 1-methylpropyl, 2 methylpropyl, 1,1-dimethylethyl, n-pentyl, 1-methylbutyl, 2-methylbutyl, 3-methylbutyl, 2,2-dimethylpropyl, 1-ethylpropyl, n-hexyl, 1,1-dimethylpropyl, 1,2-dimethylpropyl, 1-methylpentyl, 2-methylpentyl, 3-methylpentyl, 4-methylpentyl, 1,1-dimethylbutyl, 1,2-dimethylbutyl, 1,3-dimethylbutyl, 2,2-dimethylbutyl, 2,3-dimethylbutyl, 3,3-dimethylbutyl, 1-ethylbutyl, 2-ethylbutyl, 1,1,2-trimethylpropyl, 1,2,2-trimethylpropyl, 1-ethyl-1-methylpropyl, 1-ethyl-2-methylpropyl, n-heptyl, n-octyl, 2-ethylhexyl, n-nonyl, n-decyl, n-undecyl and n-dodecyl.

The alkyl chains mentioned above can be unsubstituted, hydroxylated or substituted by mercapto groups. Preferred examples which may be mentioned are hydroxymethyl, hydroxyethyl, such as [CH_3-CH(OH)- or CH_2(OH)-CH_2-] or mercaptomethyl radicals.

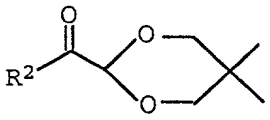
If the radical X in the formula I is $CH(OR^3)_2$, the substituents R^3 together with the oxygen atoms to which they are attached may also form a 5- or 6-membered ring. Starting materials used in this case are, for example, cyclic glyoxal monoacetals of the general formulae IIIa to IIIc.



IIIa



IIIb



IIIc

Aryl for R^1 and R^2 is to be understood as an aromatic ring or ring system having 6 to 18 carbon atoms in the ring system, for example phenyl or naphthyl, which may be unsubstituted or substituted by one or more radicals, such as halogen, for example fluorine, chlorine or bromine, cyano, nitro, amino, C_1-C_4-alkylamino, C_1-C_4-dialkylamino, hydroxyl, C_1-C_4-alkyl, C_1-C_4-alkoxy or other radicals.

Preferred radicals for R^1 are hydrogen and the branched or straight-chain C_1-C_8-alkyl chains mentioned in the list above, particularly preferably C_1-C_3-alkyl chains. Very particularly preferred radicals for R^1 are hydrogen, methyl and ethyl.

Preferred radicals for R^2 are phenyl and the branched or straight-chain C_1-C_8-alkyl chains from the list mentioned above, particularly preferably C_1-C_3-alkyl chains. A very particularly preferred radical for R^2 is methyl.

Preferred alkyl radicals for R^3 are the branched or straight-chain C_1-C_8-alkyl chains from the list mentioned above, particularly preferably C_3-C_8-alkyl chains, such as, for example, n-propyl, 1-methylethyl, n-butyl, 1-methylpropyl, 2-methylpropyl, 1,1-dimethylethyl, n-pentyl, 1-methylbutyl, 2-methylbutyl, 3-methylbutyl, 2,2-dimethylpropyl, 1-ethylpropyl, n-hexyl, n-heptyl, n-octyl or 2-ethylhexyl.

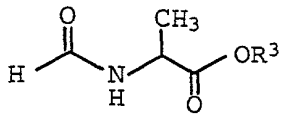
Preferred radicals for R^4 are the branched or straigh-chain C_1-C_8-alkyl chains from the list mentioned above, particularly preferably C_1-C_3-alkyl chains. Very particularly preferred radicals for R^1 are methyl, ethyl, n-propyl and isopropyl.

Depending on the amount of carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH employed, the formation of the different N-acyl derivatives of the formula I can be controlled in a targeted manner.

Thus, contrary to expectation, it has been found that reaction of an amount of carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH employed of in each case from 250 to 800 mol%, preferably from 400 to 600 mol%, based on the acetal of the formula II employed, gives N-acylamino acid esters of the formula I where X = $COOR^3$.

A particularly advantageous embodiment of the process was found to be the use of the carboxamide R^1-$CONH_2$ and the carboxylic acid R^4-COOH in identical molar proportions.

The process according to the invention is particularly suitable for preparing N-formyl-α-aminopropionic acid esters of the formula Ia

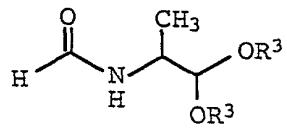


Ia

in which the substituent R^3 is C_1-C_8-alkyl, preferably C_3-C_8-alkyl.

Formation of the N-acylamino acetals of the formula I where X = $CH(OR^3)_2$ is preferred when the amount of carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH employed is in each case from 50 to 250 mol%, preferably from 100 to 200 mol%, based on the acetal of the formula II employed. In this case, too, it is particularly advantageous to employ carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH in the reaction in a molar ratio of 1:1.

In the case of the N-acylamino acetals of the formula I, the process according to the invention is advantageously suitable for preparing N-formyl-2-aminopropionaldehyde acetals of the formula Ib



Ib

in which the substituent R^3 is C_1-C_8-alkyl, preferably C_3-C_8-alkyl.

The conversion both into the N-acylamino acid esters and into the N-acylamino acetals is carried out at from 40 to 200°C, preferably from 60 to 150°C.

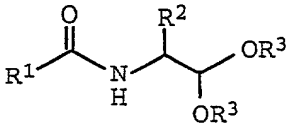
According to the invention, the reaction is carried out in a pressure range of from 200 to 1000 mbar, preferably between 500 and 1000 mbar, particularly preferably at atmospheric pressure.

The reaction can be carried out with or without additional solvent. The reaction is preferably carried out without adding a solvent.

Moreover, the process according to the invention can be carried out advantageously as a "one-pot process", giving both N-acylamino acid esters and the novel N-acylamino acetals in excellent yields.

The isolation of the desired end product is carried out in a manner known per se. In the case of liquid reaction products, the esters or acetals formed are usually purified by distillation.

The invention also provides N-acyl derivatives of the formula Ic,



Ic

in which the substituents independently of one another have the following meanings:

R^1 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^2 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^3 is C_1-C_{12}-alkyl.

Preference is given to N-acyl derivatives of the formula Ic, in which the substituents independently of one another have the following meanings:

R^1 is hydrogen, C_1-C_8-alkyl;

R^2 and R^3 are C_1-C_8-alkyl.

With respect to the exact definition of the substituents R^1 to R^3, both in the general and the preferred embodiments, the definitions given at the outset for the compound I should be referred to.

The N-acylamino acetals of the formula Ic are suitable for use as intermediates for preparing oxazoles.

The following examples are used to illustrate the subject matter of the present invention in more detail.

Example 1

Butyl N-formyl-D,L-alaninate from methylglyoxal di-n-butyl acetal.

100 g of methylglyoxal dibutyl acetal (purity 93.5%, prepared according to EP 036539) were mixed with 100 g of formamide and admixed with 100 g of formic acid over a period of 10 min. The temperature of the mixture increased to 40°C, and the mixture was then heated to reflux temperature within 20 min. After a reaction time of 2 hours, the reaction mixture, which had been cooled to room temperature, was washed with dilute sodium carbonate solution, and the desired product was distilled under reduced pressure at 2 mbar. This gave 74.5 g of pure butyl N-formyl-D,L-alaninate (93% of theory).

Example 2

2-ethylhexyl N-formyl-D,L-alaninate from methylglyoxal di-2-ethylhexyl acetal

50 g of methylglyoxal di-2-ethylhexyl acetal (purity 92%) were boiled under reflux with 30 g of formamide and 30 g of formic acid for 2.5 hours. The mixture was washed with 200 ml of sodium carbonate solution and distilled. From the main fraction, 29.8 g of 2-ethylhexyl N-formyl-D,L-alaninate (89% of theory) were isolated.

Example 3

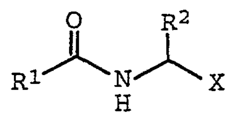
N-formylaminopropionaldehyde di-n-butyl acetal from methylglyoxal di-n-butyl acetal

100 g of methylglyoxal dibutyl acetal (purity 93.5%, prepared according to EP 036539) were mixed with 50 g of formamide and admixed with 50 g of formic acid over a period of 10 min. The

temperature of the mixture increased to 40°C, and the mixture was then heated to reflux temperature within 20 min. After a reaction time of 2 hours, the reaction mixture, which had been cooled to room temperature, was washed with dilute sodium carbonate solution, and the desired product was distilled under reduced pressure at 2 mbar. This gave 39 g of N-formylaminopropionaldehyde di-n-butyl acetal.

We claim:

1. A process for preparing N-acyl derivatives of the formula I,



I

in which the substituents independently of one another have the following meanings:

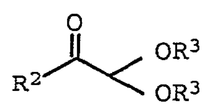
X is $CH(OR^3)_2$, $COOR^3$;

R^1 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^2 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^3 is C_1-C_{12}-alkyl,

which comprises reacting a carboxamide R^1-$CONH_2$ of the formula II with a glyoxal monoacetal derivative of the formula III,



III

in the presence of a carboxylic acid R^4-COOH of the formula IV where R^4 = C_1-C_{12}-alkyl, where the substituents R^1 to R^3 are as defined above.

2. A process as claimed in claim 1, wherein the substituents have the following meanings:

X is $COOR^3$;

R^1 is hydrogen, C_1-C_8-alkyl;

R^2 is C_1-C_8-alkyl, aryl, unsubstituted or substituted;

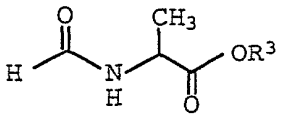
R^3 and R^4 are C_1-C_8-alkyl.

3. A process as claimed in claim 2, wherein the substituents have the following meaning:

R^1 is hydrogen;

R^2 to R^4 are C_1-C_8-alkyl.

4. A process as claimed in any of claims 2 or 3 for preparing N-formyl-α-aminopropionic acid esters of the formula Ia



Ia

in which the substituent R^3 is C_1-C_8-alkyl.

5. A process as claimed in claim 1, wherein the substituents have the following meanings:

X is $CH(OR^3)_2$;

R^1 is hydrogen, C_1-C_8-alkyl;

R^2 is C_1-C_8-alkyl, aryl, unsubstituted or substituted;

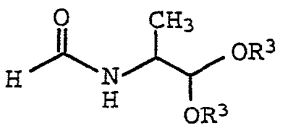
R^3 and R^4 are C_1-C_8-alkyl.

6. A process as claimed in claim 5, wherein the substituents have the following meanings:

R^1 is hydrogen;

R^2 to R^4 are C_1-C_8-alkyl.

7. A process as claimed in either of claims 5 or 6 for preparing N-formyl-2-aminopropionaldehyde derivatives of the formula Ib



Ib

in which the substituent R^3 is C_1-C_8-alkyl.

8. A process as claimed in any of claims 2 to 4, wherein the amount of the respective carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH employed is from 250 to 800 mol%, based on the acetal of the formula II employed.

9. A process as claimed in claim 8, wherein the amount of the respective carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH employed is from 400 to 600 mol%, based on the acetal of the formula II employed.

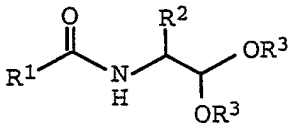
10. A process as claimed in any of claims 8 or 9, wherein the carboxamide R^1-$CONH_2$ and the carboxylic acid R^4-COOH are employed in the reaction in a molar ratio of 1:1.

11. A process as claimed in any of claims 5 to 7, wherein the amount of the respective carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH employed is from 50 to 250 mol%, based on the acetal of the formula II employed.

12. A process as claimed in claim 11, wherein the amount of the respective carboxamide R^1-$CONH_2$ and carboxylic acid R^4-COOH employed is from 100 to 200 mol%, based on the acetal of the formula II employed.

13. A process as claimed in either of claims 11 or 12, wherein the carboxamide R^1-$CONH_2$ and the carboxylic acid R^4-COOH are employed in the reaction in a molar ratio of 1:1.

14. A N-acyl derivative of the formula Ic,



Ic

in which the substituents independently of one another have the following meanings:

R^1 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^2 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^3 is C_1-C_{12}-alkyl.

15. A N-acyl-derivative as claimed in claim 14 in which the substituents independently of one another have the following meanings:

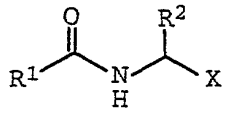
R^1 is hydrogen, C_1-C_8-alkyl;

R^2 and R^3 are C_1-C_8-alkyl.

Preparation of N-acylamino acid esters and N-acylamino acetals

Abstract

A process for preparing N-acyl derivatives of the formula I,



I

in which the substituents independently of one another have the following meanings:

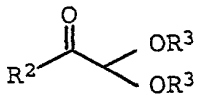
X is $CH(OR^3)_2$, $COOR^3$;

R^1 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^2 is hydrogen, C_1-C_{12}-alkyl, aryl, unsubstituted or substituted;

R^3 is C_1-C_{12}-alkyl,

which comprises reacting a carboxamide R^1-$CONH_2$ of the formula II with a glyoxal monoacetal derivative of the formula III,



III

in the presence of a carboxylic acid R^4-COOH of the formula IV where R^4 = C_1-C_{12}-alkyl, where the substituents R^1 to R^3 are as defined above,
is described.

Declaration, Power of Attorney

0050/050634

We (I), the undersigned inventor(s), hereby declare(s) that:

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Preparation of N-acylamino acid esters and N-acylamino acetals

the specification of which

[x] is attached hereto.

[ ] was filed on _____ as

Application Serial No. _____

and amended on _____.

[ ] was filed as PCT international application

Number _____

on _____

and was amended under PCT Article 19

on _____ (if applicable).

We (I) hereby state that we (I) have reviewed and understand the contents of the above–identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. § 119(a)–(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application(s)

Application No.	**Country**	**Day/Month/Year**	**Priority Claimed**	
19940641.3	*Germany*	*26 August 1999*	[x] Yes	[ ] No

We (I) hereby claim the benefit under Title 35, United States Codes, § 119(e) of any United States provisional application(s) listed below.

(Application Number)	(Filing Date)
(Application Number)	(Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

Application Serial No.	**Filing Date**	**Status (pending, patented, abandoned)**

And we (I) hereby appoint **Messrs. HERBERT. B. KEIL**, Registration Number 18,967; and **RUSSEL E. WEINKAUF**, Registration Number 18,495; the address of both being Messrs. Keil & Weinkauf, 1101 Connecticut Ave., N.W., Washington, D.C. 20036 (telephone 202–659–0100), our attorneys, with full power of substitution and revocation, to prosecute this application, to make alterations and amendments therein, to sign the drawings, to receive the patent, and to transact all business in the Patent Office connected therewith.

We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

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